TITLE: Pyrimidine derivatives
INVENTOR(S): Boon, Wm. R.
PATENT ASSIGNEE(S): Imperial Chemical Industries Ltd.
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PATENT INFORMATION:

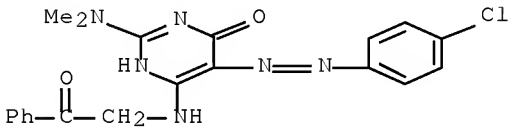
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 763041		19561205	GB 1952-9782	19520418 <--

GI For diagram(s), see printed CA Issue.

AB N:C(NXY).N:CW.C(N:NAr):CZ (I), useful as intermediates in the preparation of compds. active against schistosomiasis, were prepared [MeNC(:NH)NH2]2.H2SO4 91 refluxed 30 min. with a solution of MeONa (prepared from Na 15 and MeOH 300), CH2(CO2Et)2 116 added, the mixture heated a further 6 hrs., H2O 450 patts added together with sufficient AcOH to render the solution acid to litmus, and the precipitate filtered off gave N:C(NMe2).N:C(OH).CH:COH (II). II 155 and POCl3 100 refluxed 30 min., the mixture cooled, poured into ice 800 and 32% aqueous NaOH 330 parts, the precipitate filtered off, washed, and purified by steam distillation gave the 4,6-Cl2 analog (III), m. 54°. III 38 and alc. NH3 100 patts heated 18 hrs. at 120°, the mixture cooled, steam distilled, and the residue filtered off gave N:C(NMe2).N:C(NH2).CH:CCl (IV), m. 151°. To IV 43 parts in AcOH 400 parts and H2O 1000 parts was added a solution of p-ClC6H4N2Cl (V) (prepared by diazotization of 4-ClC6H4NH2) and sufficient NaOAc to make the solution neutral to Congo red, the solution let stand 17 hrs., and the precipitate filtered off to give I (X = Y = Me, Z = NH2, Ar = 4-ClC6H4, W = Cl), m. 228°. Similarly were prepared the following I by coupling with N:C(NMe2).N:C(NHMe).CH:CCl (m. 78°) (X = Y = Me, Z = NHMe, and W = Cl in all cases) (Ar and m.p. given): 4-ClC6H4, 184°; Ph, 163°; 2-MeOC6H4, 174°; 4-O2NC6H4, 265°; 1-naphthyl, 236°. In the same way, I (X = Y = Me, Z = NMe2, Ar = 4-ClC6H4, W = Cl), m. 91°, was prepared from N:C(NMe2).N:C(NMe2).CH:CCl, m. 53°. BzCH2(NH2)Ph and N:CCl.N:CCl.CH:CCl (VI) gave 2,4-dichloro-6-desylaminopyrimidine (VII), m. 162°. VII and alc. Me2NH refluxed 3 hrs. gave the 2-Me2N analog (VIII), m. 182°. VIII coupled with V afforded I (X = Y = Me, Z = BzPhCH, W = Cl, Ar = 4-ClC6H4), m. 253° (decomposition). Similarly were prepared from BzCH(NH2)C6H4Cl-4.HCl (m. 248°): α-(4- chlorophenyl-α-(2,4-dichloro-6-pyrimidylamino)acetophenone, m. 144-5°; α-(4-chlorophenyl)-α-(4-chloro-2-dimethylamino- 6-pyrimidylamino)acetophenone, m. 155-6°; α-(4-chlorophenyl)- α-4-chloro-5-p-chlorophenylazo-2-dimethylamino-6-pyrimidylamino)acetophenone, m. 248°. III and H2NCH2CO2Et refluxed 36 hrs. in alc. gave the 4-EtO2CCH2NH analog, m. 121°, which on coupling with V gave I (X = Y = Me, Z = EtO2CCH2NH, W = Cl, Ar = 4-ClC6H4), m. 214°. III and NaOMe solution stirred 18 hrs. gave the 4-MeO analog (IX), m. 62°. IX and 10N HCl heated 30 min. on a steam bath afforded the 4-HO analog (X), m. 216°. X coupled with V yielded I (X = Y = Me, Z = HO, W = Cl, Ar = 4-ClC6H4), m. 222°. N:CCl.N:CCl.CH:CNHMe and Et2NH in MeOH refluxed 8 hrs. gave the 2-Et2N derivative, m. 39-40°, which on coupling with V afforded I (X = Y = Et, Z = NHMe, Ar = 4-ClC6H4, W = Cl), m. 126°. Similarly were prepared: 4-chloro-6-methylamino-2-piperidinopyrimidine, m. 117°, and its 5-p-ClC6H4N:N derivative, m. 190°. H2NCH2C(:NNHCONH2)Me and VI treated with NaOEt solution gave 2,4-dichloro-6-pyrimidylaminoacetone (XI) semicarbazone (XII), m. 209°. XII heated with 2N HCl gave XI, m. 102°. XI and Me2NH in EtOH refluxed 3 hrs. yielded the 2-Me2N analog, m. 134°, which on coupling with V gave I (X = Y = Me, Z = AcCH2NH, Ar = 5-p-ClC6H4, W = Cl), m. 233°.

IT 103388-37-0
(Derived from data in the 6th Collective Formula Index (1957-1961))

RN 103388-37-0 ZCAPLUS
CN Acetophenone, 2-[[5-(p-chlorophenylazo)-2-dimethylamino-6-hydroxy-4-pyrimidinyl]amino]- (6CI) (CA INDEX NAME)



IT 103387-84-4P, Acetophenone, 4'-chloro-2-[[5-(p-chlorophenylazo)-2-dimethylamino-6-hydroxy-4-pyrimidinyl]amino]- 103757-94-4P, Acetophenone, 2-[[6-chloro-5-[p-chlorophenylazo)-2-dimethylamino-4-pyrimidinyl]amino]-2-(p-chlorophenyl)- 103758-00-5P, Acetophenone, 2-[[6-chloro-5-[p-chlorophenylazo)-2-dimethylamino-4-pyrimidinyl]amino]-2-phenyl- 103758-01-6P, Acetophenone, 4'-chloro-2[[5-(p-chlorophenylazo)-2-dimethylamino-6-hydroxy-4-pyrimidinyl]amino]-2-phenyl- 109694-08-8P, Acetophenone, 2-[(6-chloro-2-dimethylamino-4-pyrimidinyl)amino]-2-phenyl- 109804-94-6P, Acetophenone, 2-[(6-chloro-2-dimethylamino-4-pyrimidinyl)amino]-2-(p-chlorophenyl)-
RL: PREP (Preparation)
(preparation of)

RN 103387-84-4 ZCAPLUS
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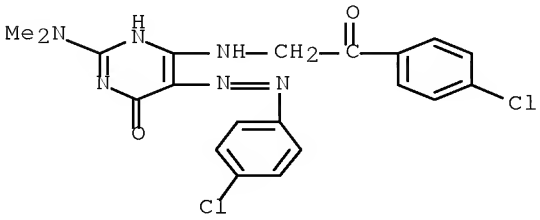

RN 103757-94-4 ZCAPLUS
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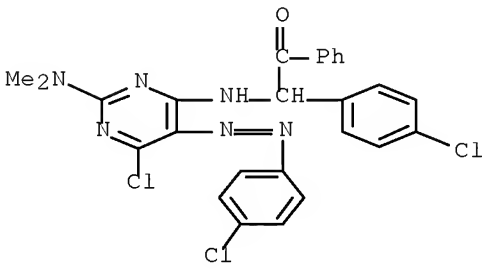

RN 103758-00-5 ZCAPLUS
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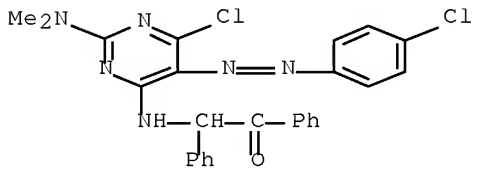

RN 103758-01-6 ZCAPLUS
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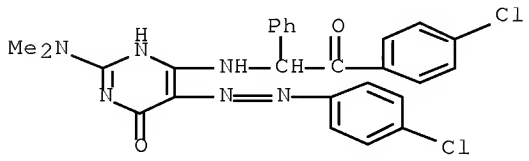

RN 109694-08-8 ZCAPLUS
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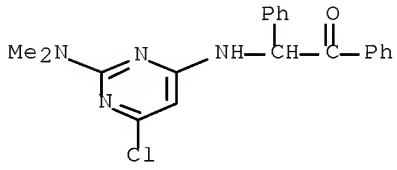

RN 109804-94-6 ZCAPLUS
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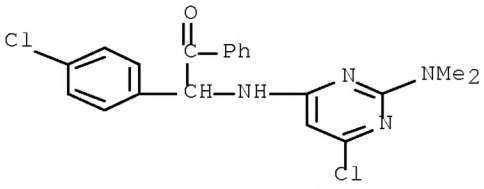
O
Cl
C
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CH
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